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INITIAL CORROSION TESTS ON BRUSH PROCESS QM AND QRM BERYLLIUM

PART 1-A

INTERIM REPORT ON THE CORROSION OF BERYLLIUM IN SIMULATED COOLING WATER FOR THE MATERIALS TESTING REACTOR

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REACTOR TECHNOLOGY DIVISION
Section IV - Engineering Materials

INITIAL CORROSION TESTS ON BRUSH PROCESS QM AND QRM BERYLLIUM
(April 1949 through September 1949)

Part I-A

Interim Report on the Corrosion of Beryllium in Simulated Cooling Water for the Materials Testing Reactor

Arnold R. Olsen

Date Issued
JUL 14 1950

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TABLE OF CONTENTS

		Page
1.0	INTRODUCTION	5
2.0	ABSTRACT	5
3.0	CORROSION TESTING CONDITIONS	6
4.0	TEST MATERIALS	6
	4.1 Types of Materials	6
	4.2 History of Test Materials	7
	4.3 Metallurgical Data	10
	4.31 Beryllium (Hardness-Tensile Strengths)	10
	4.32 2S Aluminum	16
	4.33 356 Aluminum	16
	4.4 Preparation and Defilming of Test Specimens	16
5.0	CORROSION TESTING METHODS	16
	5.1 Test Apparatus	16
	5.2 Types of Tests	17
	5.3 Solution Control	17
6.0	CORROSION DATA ON "QM" AND "QRM" BERYLLIUM IN THE AS MACHINED CONDITION	17
	6.1 Effect of Low Hydrogen Peroxide Concentrations	17
	6.2 Effect of High Hydrogen Peroxide Concentrations	18
7.0	CORROSION DATA ON "QM" AND "QRM" BERYLLIUM IN THE ANNEALED CONDITION	19
	7.1 Effect of Low Hydrogen Peroxide Concentrations	19
	7.2 Effect of High Hydrogen Peroxide Concentrations	20
8.0	CORROSION DATA ON "QM" AND "QRM" BERYLLIUM IN CONTACT WITH OTHER METALS	22
	8.1 Object of Bi-metallic Galvanic Tests	22
	8.2 Beryllium - 2S Aluminum Couples	22
	8.3 Beryllium - 356 Aluminum Couples	23

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Page

8.31 Effect of Low Hydrogen Peroxide Concentrations 24
8.32 Effect of High Hydrogen Peroxide Concentrations 25

9.0 THE EFFECTS OF POOR DEMINERALIZED WATER 26

9.1 Fabrication History and Chemical Analysis of Lot 5 QRM Beryllium 26
9.2 Corrosion Data on QRM Beryllium in Y-12 Demineralized Water 30

10.0 INTERPRETATION OF CORROSION DAMAGE 31

11.0 DISCUSSION OF RESULTS 31

12.0 FUTURE PROGRAMS 32

13.0 ACKNOWLEDGMENTS 33

1.0 INTRODUCTION

This is an interim report covering initial corrosion tests in beryllium metal from Brush Beryllium Corporation's new powder techniques, QM and QRM. Most of the data in this report have previously appeared in various ORNL Technical Division Progress and Quarterly Reports.

The urgency of finding a suitable moderating and reflecting material for the Materials Testing Reactor has led to the choice of as short a corrosion testing period as possible to still give reasonably reliable information. Consequently, all tests reported here have been of sixty day duration. Low temperature, long term tests, and high temperature, high pressure accelerated corrosion tests are now in operation; these will be reported in the near future.

2.0 ABSTRACT

This is Part I-A of a series of reports covering the corrosion resistance of various metals and alloys to be used in the construction of the Materials Testing Reactor.

A more detailed discussion of the problem, testing conditions, and the mechanisms of corrosion can be found in ORNL-298 "Interim Report on the Corrosion of Beryllium in Simulated Cooling Water for the Proposed Development Reactor", and no attempt will be made to repeat this here.

Static corrosion tests were conducted for 60 days in demineralized water containing small amounts of hydrogen peroxide at 85° C in the pH range 5.5 to 6.5. Samples of QM and QRM beryllium, produced by two recently developed powder process techniques, were tested.

The data provided by these experiments indicate that in all cases the metal from these two processes has corrosion resistance comparable with the best vacuum cast and extruded metal tested by this group. QRM metal seems to be slightly superior to the QM metal under all conditions. A calculated maximum penetration rate of 0.0014 inches per year was observed.

Since, during the period of these tests, the group moved to new quarters and as a consequence changed its source of supply of demineralized water, one group of tests shows the importance of maintaining the water as pure as possible. Tests are being started to determine the maximum allowable

2.0 ABSTRACT (Con't.)

concentration of various ionic constituents in the demineralized water. The results of all tests show that annealing after machining decreases the corrosion resistance of the beryllium at least in the initial stages of attack.

3.0 CORROSION TESTING CONDITIONS

For ready reference, a brief summary of the conditions under which this metal was tested is given here:

Medium - - - - - - - - - - - - Deaerated and demineralized water

Specific resistance: 1-2 x 15^5 ohms/cm at 25° C

Maximum ion concentrations:

Sodium 1-2 ppm

Aluminum 0.05 ppm

Iron 0.05 "

Chloride 0.20 "

Temperature - - - - - - - - - - 80 - 95° C

pH - - - - - - - - - - - - - - 5.5 - 6.5

H_2O_2 - - - - - - - - - - - - - 0.005M and 0.0005M

4.0 TEST MATERIALS

4.1 Types of Material

As was originally reported, the available supply of beryllium metal was limited and the techniques and processes of fabrication were and are in the experimental stage. While the major portion of the effort has been to develop a reliable vacuum cast and extruded metal;

4.1 Types of Material (Con't.)

the Brush Beryllium Corporation has continued to work on the improvement of their powder process. This portion of the report deals with the corrosion resistance of QM and QRM metal produced by their latest processes.

Only two metals were used to test the galvanic couple effects; 356 aluminum and 2S aluminum.

4.2 Histories of Test Materials

The only available information on the production of the metal which is used to produce the powder for compacting is that contained in Mr. J. L. Gregg's memorandum, Central Files Number 49-4-144, "Brush Hot-Pressed Beryllium for Corrosion Tests."

In this memorandum the metal is listed as Lots 1, 2, 3 and 4 with their respective means of production and chemical analysis. For convenience this listing is quoted here.

METAL TO BE TESTED
Lot 1

"Consists of 12 pieces, each 1" x 4" x 6" received several weeks ago. These pieces were cut from three large pieces of the following analyses:

	Y-4381BPQM	Y-4382BPQM	Y-4383BPQM
BeO	1.02%	1.86 %	1.22 %
Fe	0.13%	0.118%	0.128%

The average analysis of the powders used was:

Assay	99%
Al	0.18%
Fe	0.11%
Mg	0.05%

The metal used in producing the powder was QM metal, which is made by melting the raw metal in an open pot in a high-frequency furnace together with a flux containing the fluorides of calcium, magnesium and beryllium. This flux extracts the slag and magnesium, but tends to add a small amount of calcium. The hot pressing was done in a large furnace where the pressure was applied by means of a plunger.

4.2 Histories of Test Materials (Con't.)

Lot 2

This is a single bar approximately 1" x 2" x 44" made by the envelope technique and sintered under a pressure of 150 p.s.i. The powder was made from pressure-furnace remelt, the flux for remelting being a mixture of magnesium and beryllium fluoride. The remelting was made before the technique was completely worked out and the magnesium content is high, as shown by the analyses below. Results of corrosion tests on this bar should indicate whether or not magnesium influences corrosion.

National Spectrographic Labs - Spectrographic

Ag	0.5	ppm (about)
Al	2000	ppm
Ca	30 to 50	ppm
Cd	-0.2	ppm
Cr	94	ppm
Cu	140	ppm
Fe	1150	ppm
Mg	1750	ppm
Mn	113	ppm
Na	36	ppm
Ni	83	ppm
Si	725	ppm
Sn	-1	ppm
Zn	39	ppm
Li	-0.3	ppm
Pb	25	ppm (about)

Brush Beryllium Co. - Chemical

BeO	1.21%	
Slag	0.20%	
Assay	98.63%	
Fe	1090	ppm
Al	988	ppm
Mg	2760	ppm
Ni	134	ppm
Cr	131	ppm
Si	386	ppm

4.2 Histories of Test Materials (Con't.)

Lots 3 and 4

These lots are bars numbers 6 and 7, respectively. These bars are supposedly alike. They are 1/2" x 1 15/16" x 41 1/2" as rough machined. They were machined with a milling cutter and the results of considerable chatter are obvious. Brush was advised that the surface of all samples used for corrosion tests cut from these bars would be machined at Oak Ridge. These bars were made by the envelope technique, using a pressure of 300 p.s.i. The metal was refined by the same process as the metal for Lot No. 2, but the process was supposedly under better control. Chemical analyses were as follows:

National Spectrographic Labs. - Spectrographic

	Bar 6	Bar 7	
Ag	0.5 about	0.6 about	ppm
Al	1200	1400	ppm
Cd	-0.2	-0.2	ppm
Co	-1	-1	ppm
Cu	130	83	ppm
Fe	1050	855	ppm
Li	-0.3	-0.3	ppm
Mg	2500	1675	ppm
Mn	108	97	ppm
Ni	55	55	ppm
Si	560	410	ppm
Zn	60	60	ppm

Brush Beryllium Co. - Chemical

	Bar 6	Bar 7	
Be	99.18 %	99.7 %	
BeO	0.89 %		
Slag	0.04 %		
Fe	0.095%	0.092 %	
Al	0.08 %	0.09 %	
Mg	0.06 %	0.2 %	being checked
B	0.06 %	1.2 ppm	
Density	1.862	1.86 +	being checked

4.3 Metallurgical Data

4.31 Beryllium

The majority of the beryllium was tested in the as-machined-condition. Those samples which were annealed after machining were degreased and heated in a vacuum of 100 μ to 830° C and held for one hour. They were then allowed to cool in the furnace and under vacuum.

Hardness

Hardness measurements on the various Lots in the "as machined and annealed condition" are listed in Table 1., together with comparative values on various extruded metals and the Brush Q Process beryllium.

Tensile Strength

Tensile specimens were machined from two billets of Lot 1, QM beryllium. Two specimens were used for control samples, while the other six were exposed for 60 days to different concentrations of hydrogen peroxide solution at 85° C. Figure 1 is a scale drawing of the tensile specimen used. Table 2 gives the corrosion data as well as the ultimate tensile strengths of the specimens when tested after corrosion.

The results of this series of tests indicate that exposure to simulated MTR cooling water has little effect on the tensile properties of the beryllium. These results further verify the conclusion, stated in ORNL-298, that the corrosion attack on beryllium is not of an intergranular nature.

Figures 2 and 3 are pictures of the specimens after they have been broken in tension. Pictures of specimens 4382-8 and 4383-III have not been included as they are essentially the same as 4382-7 and 4383-VIII, respectively. The black circle at the fracture (Figure 3 - IV) surrounds a corrosion pit.

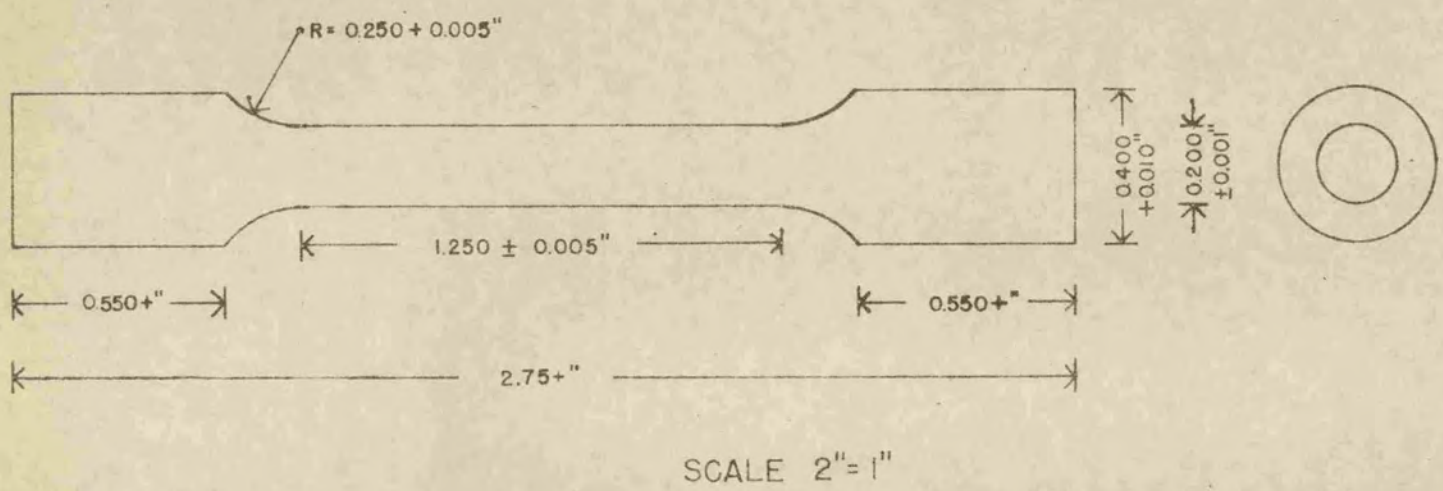
TABLE 1

HARDNESS OF BERYLLIUM SPECIMENS

Identification	Hardness			
	As Machined		Annealed	
	R 15T	Equiv. R_B	R 15T	Equiv. R_B
Lot 1	83.5	70	81.5	64
Lot 2	87	81	85.5	77
Lot 3	85	75	84	72
Lot 4	87.5	83	84.5	73
Vacuum cast and extruded Beryllium*		59		57
Q Process Beryllium*		70		69
Vacuum cast and extruded Beryllium**	84.5	74		
Q Process Beryllium**	82.5	67		

* These are the values reported in CT 3650 "Comparison of the Brush Process "Q" Beryllium with Extruded Cast Beryllium, Extruded Flake, and Extruded Turnings," Table IV Page 3.

** These are values previously reported in ORNL-298, Table II, page 14.



SKETCH OF TENSILE SPECIMEN

MAY BE MACHINED ON CENTERS

FIG. 1

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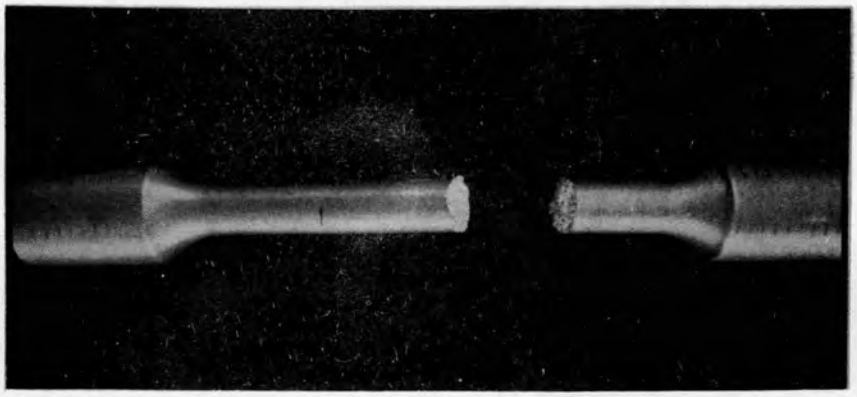
DWG. 9103

PHOTO 6537



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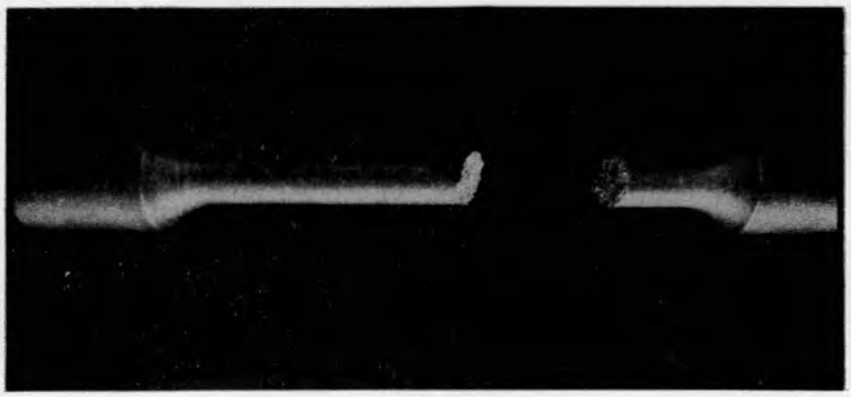

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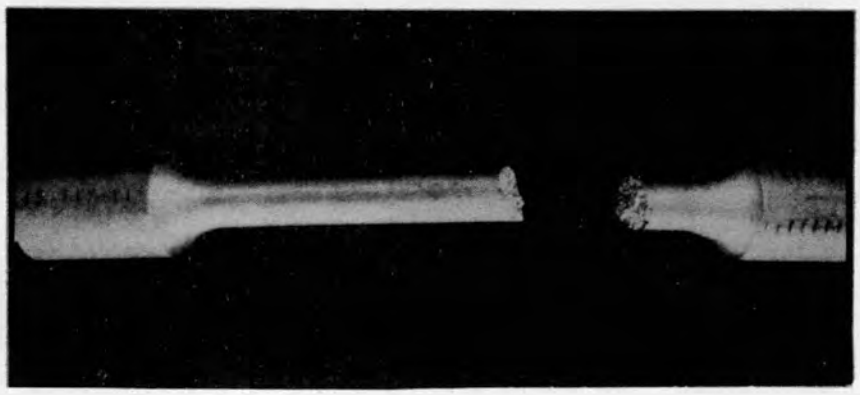

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FIGURE 2

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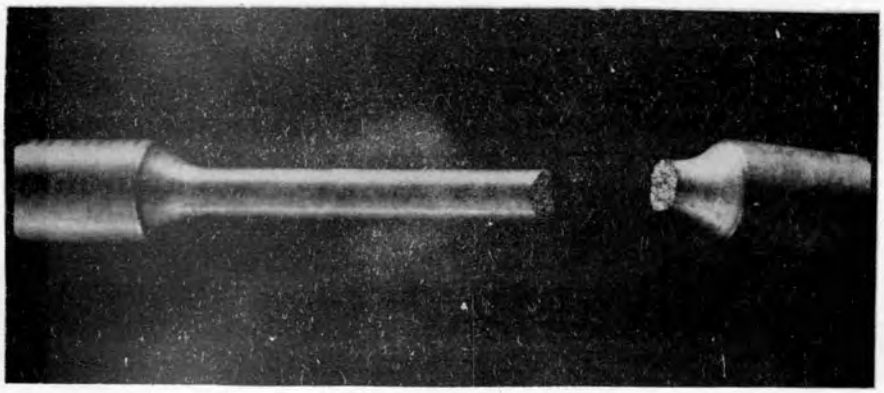
QM Beryllium Tensile Strength
Billet 4382

PHOTO 6536



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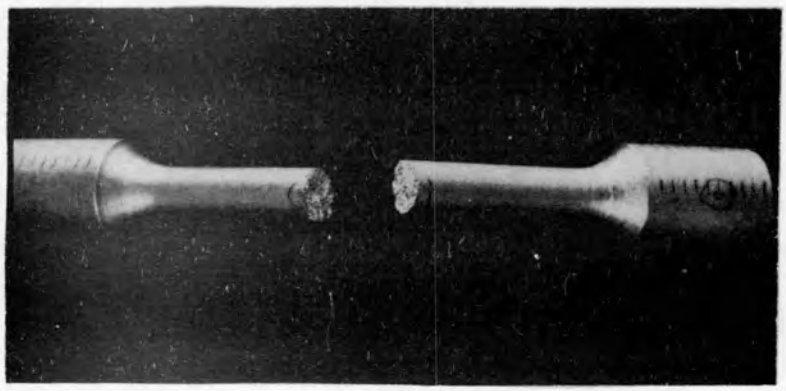

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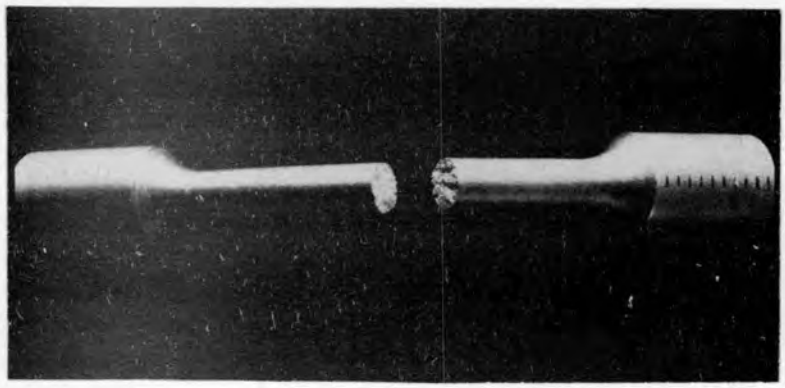

VIII

Magnification 1.4X

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FIGURE 3

QM Beryllium Tensile Specimens
Billet 4383

[redacted]

TABLE 2

CORROSION AND TENSILE DATA ON QM BERYLLIUM EXPOSED TO SIMULATED MTR COOLING WATER AT 85°C FOR 60 DAYS

Billet No.	Specimen	Pit Count per sq.cm.	Max. Pit depth(mils)	Penetration Rate mils/mo.	Weight Loss (M D M)	Ultimate Tensile Strength (psi)	Description of break
4382-7*	Longitudinal	neg.	neg	.010	4.5	27,360	clean
4382-3	Transverse	0.2	0.8	.020	9.3	30,420	Parallel Fracture
4382-6	Longitudinal	0.3	1.6	.018	8.8	28,850	clean
4382-8	Longitudinal					28,070	clean
4383-III*	Transverse					30,900	clean
4383-II	Transverse	1.2	0.8	.023	10.6	31,660	clean
4383-IV	Longitudinal	0.4	1.2	.025	11.7	31,540	Initiated at Corrosion pit
4383-VIII	Longitudinal	neg.	neg.	.016	7.4	35,670	clean

* These were uncorroded control samples.

[redacted]

4.3 Metallurgical Data (Con't.)

4.32 2S Aluminum

All 2S aluminum samples were annealed in air at 425° C after machining.

4.33 356 Aluminum

Aluminum alloy No. 356 was subjected to T-7 heat treatment before exposure:

1) Heat 12 hours at 538° C

2) Quench in boiling water

3) Stabilize at 227° C for 9 hours

4.4 Preparation and Defilming of Test Specimens

All specimens were machined to the desired dimensions, degreased in a vapor degreaser and heat treated before the total surface areas were determined.

All beryllium specimens were defilmed prior to testing in 25% nitric acid for 20 minutes at room temperature. All aluminum specimens were defilmed before exposure in 2% hydrofluoric acid for 30 seconds. These chemical defilming treatments are used to remove the existing oxide layers and expose fresh metal to the corrodent.

After removal from the corrosion test solutions, all samples were defilmed in a mixture of 2% chromic acid and 5% phosphoric acid for 20 minutes at 50° C.

5.0 CORROSION TESTING METHODS

5.1 Test Apparatus

The apparatus is the same as that described and pictured in ORNL 298. Briefly, this consists of 5 liter, 3 necked distilling flask supported on a transite box containing a 375 watt infra-red bulb for heating. The temperature was controlled within ± 0.5° C by a Merck-to-Merck thermoregulator-relay system. Reflux condensers prevented solution loss; hydrogen peroxide additions were made from burettes installed on each test.

5.2 Types of Test

All tests were conducted under static conditions, i.e., there was no deliberate agitation. However slight convection currents, due to heating, prevented the solutions around the specimens from being completely stagnant.

Monometal tests were conducted by individually suspending each specimen from a glass hook.

Galvanic couples were made by bolting a beryllium sample and an aluminum sample together using nuts and bolts machined from heat treated aluminum. This assembly was then supported in the solution by means of glass hooks.

All tests were run in duplicate, and in order to maintain exactly the same conditions, the two samples were suspended separately in one flask.

5.3 Solution Control and Maintenance

The solutions were changed weekly to remove insoluble corrosion products and limit the depletion of any specific ionic constituent in the demineralized water.

Daily pH checks were made and adjusting was done by the use of dilute sodium hydroxide or dilute nitric acid.

Exact control of the hydrogen peroxide concentration was impossible. However by making regular additions of 4% hydrogen peroxide every 3 hours, and running daily determinations, the concentrations were maintained within reasonably close limits.

Information as to the effects of metallic ions, pH, and temperature on the stability of hydrogen peroxide solutions may be found in ORNL-298.

6.0 CORROSION DATA ON "QM" AND "QRM" BERYLLIUM IN THE AS MACHINED CONDITION

6.1 Effect of Low Hydrogen Peroxide Concentrations

Duplicate samples of Lots 1 and 2 and one sample each of Lots 3 and 4 were exposed to solutions containing 0.0005M H_2O_2 for a period of 60 days (1440 hours). The results of these tests are listed in Table 3.

6.1 Effect of Low Hydrogen Peroxide Concentrations (Con't.)

TABLE 3

STATIC CORROSION OF QM AND QRM BERYLLIUM IN THE AS-MACHINED CONDITION IN 0.0005M H_2O_2 SOLUTIONS AT 85°C

Lot No.	Pit Count per sq. cm.	Max. Pit Depth (mils)	Penetration Rate (mil/mo)	Wt. Loss (MDM*)
1	1.6	0.6	0.023	10.8
1	0.1	2.8	0.026	12.3
2	2.0	3.2	0.011	5.2
2	neg.	neg.	0.014	6.3
3	0.6	6.3	0.019	9.1
4	0.6	4.0	0.018	8.4

* MDM here and in all succeeding tables will stand for Milligrams per square decimeter per month.

The average corrosion rate of all these specimens is below that of vacuum cast and extruded berullium with the exception of Lot No. 1 which is on a par.

The surfaces of all samples after defilming were clean under microscopic examination, and there were no signs of excessive local attack on any specimen.

6.2 Effect of High Hydrogen Peroxide Concentrations

Test results on specimens of all four lots exposed to demineralized water containing 0.005M H_2O_2 at 85° C for a period of 60 days are included in Table 4.

6.2 Effect of High Hydrogen Peroxide Concentrations (Con't.)

TABLE 4

STATIC CORROSION OF QM AND QRM BERYLLIUM IN THE AS MACHINED CONDITION IN 0.005M H_2O_2 SOLUTIONS AT 85° C.

Lot No.	Pit Count per sq. cm.	Max. Pit Depth (mils)	Penetration Rate (mil/mo)	Weight Loss (M D M)
1	1.3	2.0	0.023	10.8
1	neg.	neg.	0.019	9.2
2	one pit	6.4	0.015	6.9
2	neg.	neg.	0.016	7.3
3	0.8	2.8	0.016	7.7
4	2.2	5.8	0.019	8.8

7.0 CORROSION DATA ON "QM" AND "QRM" BERYLLIUM IN THE ANNEALED CONDITION

7.1 Effect of Low Hydrogen Peroxide Concentrations

Beryllium specimens which had been annealed in vacuum for one hour at 830° C were tested to see what effect the cold working of machined specimens had on their corrosion resistance.

Test results on annealed specimens exposed to 0.005M H_2O_2 solutions at 85° C for 60 days are included in Table 5.

7.1 Effect of Low Hydrogen Peroxide Concentrations (Con't.)

TABLE 5

STATIC CORROSION OF ANNEALED QM AND QRM BERYLLIUM IN 0.0005M H_2O_2 SOLUTIONS AT 85° C

Lot No.	Pit Count per sq.cm.	Max. Pit Depth (mils)	Penetration Rate (mil/mo)	Weight Loss (MDM)
1	1.5	4.0	0.020	9.2
1	0.2	4.4	0.022	10.5
2	9.4	4.9	0.016	7.4
2	3.5	1.4	0.016	7.4
3	2.0	7.2	0.025	11.6
4	1.1	3.0	0.025	11.6

These data indicate that annealing tends to decrease the corrosion resistance of the metal, particularly with regard to pitting susceptibility.

7.2 Effect of High Hydrogen Peroxide Concentration

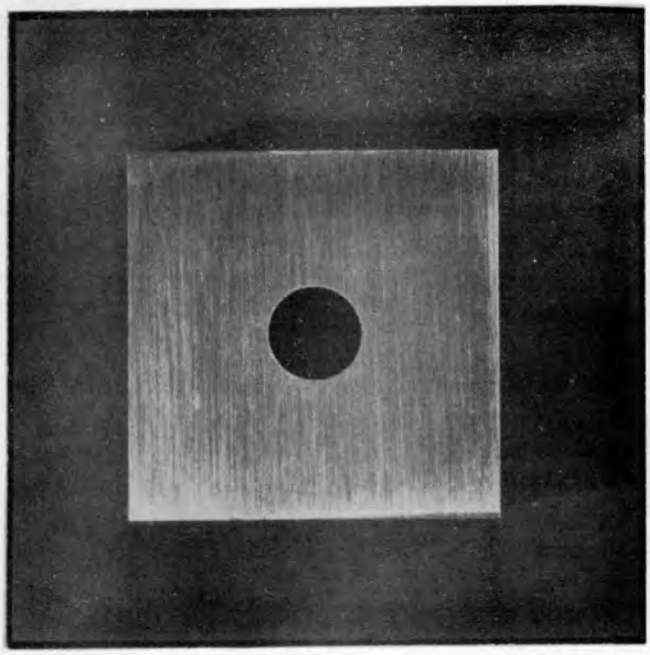
Corrosion data on annealed specimens exposed for 60 days at 85° C to demineralized water containing 0.005M H_2O_2 are included in Table 6.

TABLE 6

STATIC CORROSION OF ANNEALED QM AND QRM BERYLLIUM IN 0.005M H_2O_2 SOLUTIONS AT 85°C

Lot No.	Pit Count per sq. cm.	Max. Pit Depth (mils)	Penetration Rate (mil/mo)	Weight Loss (MDM)
1	4.4	1.6	0.024	11.1
1	1.5	2.4	0.021	9.8
2	5.5	2.4	0.025	11.9
2	12.5	2.8	0.033	15.6
3	0.7	4.8	0.021	9.9
4	8.1	2.2	0.019	8.9

PHOTO 6539



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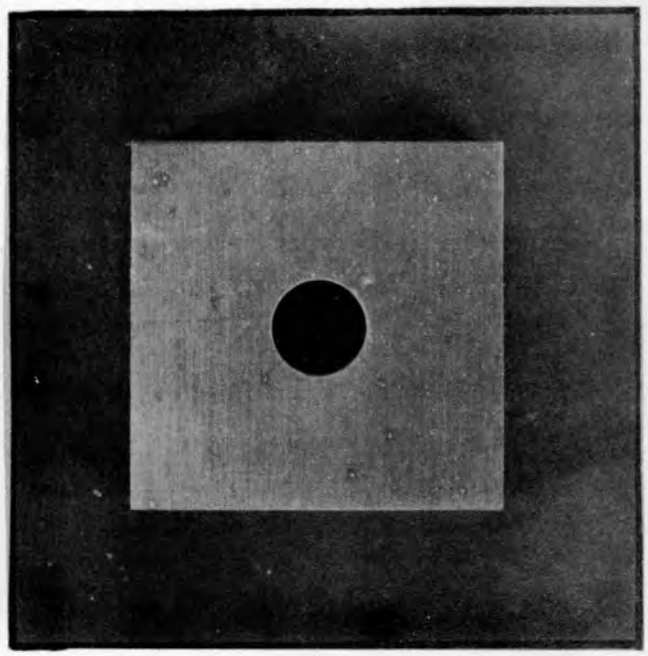

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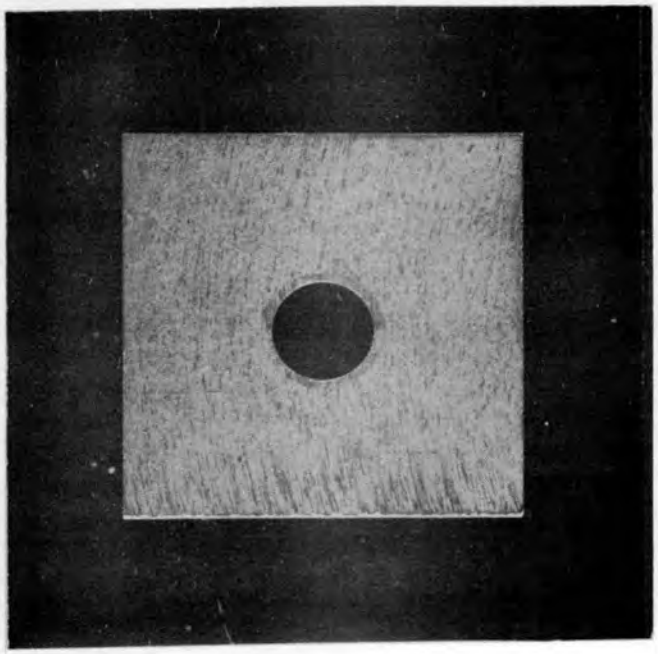

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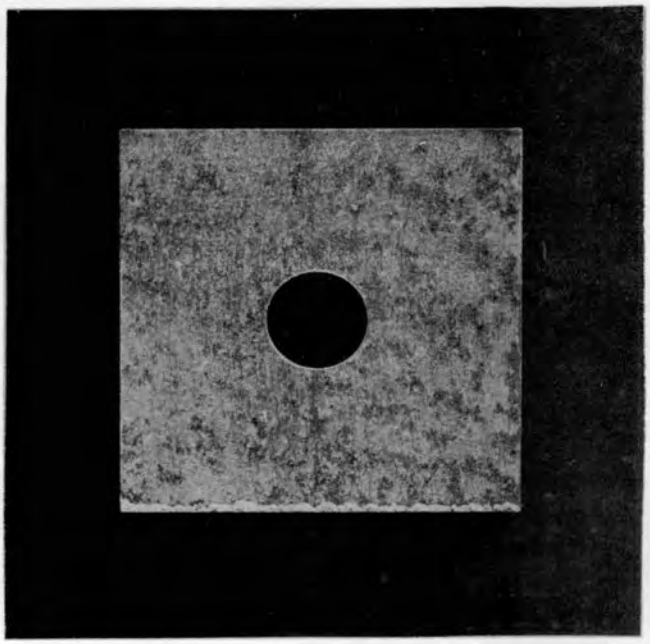

No. 4

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FIGURE 4

Effects Of Annealing On The Corrosion Resistance Of QRM Beryllium

7.2 Effect of High Hydrogen Peroxide Concentration (Con't.)

The effects of annealing on the resistance of QM and QRM beryllium, exposed to the higher concentrations of hydrogen peroxide, seem, in general, to parallel those for exposure to lower concentrations. However, it will be noted that in the case of Lot 2 specimens, the overall corrosion rate for annealed specimens is twice that of the as machined samples.

Figure 4 shows samples of Lot 2 QRM beryllium in the as-machined and machined-annealed condition. No 1 is a photograph of the sample in the as machined condition prior to corrosion testing; No. 2 is a sample which has been annealed one hour at 830° C in a vacuum of 30 μ.

The difference in corrosion susceptibility to 85° C water containing 0.005M H_2O_2 is apparent from No. 3, and No. 4. No. 3 is the as-machined sample and No. 4 the machined-annealed sample, after corroding.

In view of the apparent protective influence of the cold worked surfaces on the as machined specimens, a question of permanence arises. In order to determine whether or not this protective influence will be removed by corrosion over a long period of time, planned interval tests of one year's duration have been started.

8.0 CORROSION DATA ON "QM" AND "QRM" BERYLLIUM IN CONTACT WITH OTHER METALS

8.1 Object of Bi-metallic Galvanic Tests

Since the removable beryllium assemblies will be fitted with end boxes of 356 aluminum alloy and a major portion of the reactor core will be 2S aluminum in one form or another, bi-metallic couple tests with these alloys were conducted.

The intensity of attack on either of the metals in such a couple, is dependent on the electromotive potential developed in the solution between the two metals and the conductivity of the solution. The specific resistance of the demineralized water is therefore maintained as high as possible.

8.2 Beryllium-2S Aluminum Couples

Table 7 gives the data for corrosion tests conducted with beryllium and 2S aluminum couples in 0.005M H_2O_2 at 85° C for a period of 60 days.

8.2 Beryllium-2S Aluminum Couples (Con't.)

TABLE 7

STATIC CORROSION OF QM BERYLLIUM-
2S ALUMINUM COUPLES IN 0.005M H_2O_2 SOLUTIONS AT 85°C.

Material	Pit Count per sq. cm.	Maximum Pit Depth (mils)	Penetration Rate (mil/mo)	Weight Loss (MDM)
Be Lot 1	0.4	2.8	0.113	53.2
2S Al.	0.5	40.0	0.003	4.0
Be Lot 1	0.8	0.8	0.045	21.5
2S Al.	neg.	neg.	0.006	8.0
Be Lot 2	3.6	4.0	0.016	7.7
2S Al.	1.3	2.8	0.008	12.0
Be Lot 2	1.8	0.8	0.018	8.6
2S Al.	2.2	0.8	0.009	12.5

These data show that all specimens of QM and QRM beryllium tested were anodically attacked when coupled with 2S aluminum under these test conditions. This is similar to the behavior of extruded beryllium - 2S aluminum couples in the low peroxide (0.0005M) solutions reported in ORNL-298, but a reversal in order of the reported tests in the high (0.005M) peroxide solutions. This reversal of polarity from test to test is to be expected where the two metals have so nearly identical electrode potentials.

The rate of attack of coupled Lot 1 specimens was higher than for individual specimens tested under similar conditions. The same comparison for samples from Lot 2 shows that the 2S aluminum, while remaining the cathodic member of the couple, did tend to retard the overall corrosion rate of the beryllium.

There is no marked change in pitting frequency, however this effect is difficult to evaluate on pressed compacts where the number of voids is large and varies from sample to sample.

8.3 Beryllium - 356 Aluminum Couples

As all the 356 aluminum proposed for use in the reactor will be in the T-7 heat treated condition, samples used in these investigations have been so treated.

8.3 Beryllium - 356 Aluminum Couples (Con't.)

8.31 Effect of Low Hydrogen Peroxide Concentrations

Corrosion test results on galvanic couples of QM and QRM beryllium and 356 aluminum are reported in Table 8. These tests were conducted in 0.0005M H_2O_2 solutions at 85° C for 60 days.

TABLE 8

STATIC CORROSION OF "QM" AND "QRM" BERYLLIUM - 356 ALUMINUM COUPLES IN 0.0005M H_2O_2 SOLUTIONS AT 85° C

Material	Pit Count per sq. cm.	Maximum Pit Depth (mils)	Penetration Rate (mils/Mo)	Weight Loss (MDM)
Be Lot 1	I*	.80	0.046	21.8
356 Al.	I	neg	0	0
Be Lot 1	0.3	3.2	0.044	21.2
356 Al.	1.2	1.2	0.009	6.3
Be Lot 2	4.3	4.7	0.016	6.1
356 Al.	5.3	3.6	0.009	5.9
Be Lot 2	3.5	2.0	0.017	8.2
356 Al	2.8	2.0	0.007	4.9

*I - Innumerable - i.e. the contact surface of the specimens were covered with small pits generally so shallow as to prevent distinguishing one from another and thus render accurate counting impossible.

The results of these tests show a marked increase in the overall corrosion rate of beryllium from Lot 1 with no apparent change in this rate for Lot 2. However the pitting frequency on samples of Lot 1 remained the same for both monometal and 356 aluminum couple tests while there is an apparent increase for samples of Lot 2.

In all cases the beryllium was anodically attacked as was the case with extruded beryllium.

8.3 Beryllium - 356 Aluminum Couples (Con't.)

8.32 Effect of High Hydrogen Peroxide Concentrations

Table 9 contains the corrosion data on "QM" and "QRM" beryllium 356 aluminum couple tests in 0.005M H_2O_2 solutions at 85° C of 60 day duration.

TABLE 9

STATIC CORROSION TESTS ON "QM" AND "QRM" BERYLLIUM 356 ALUMINUM COUPLES IN 0.005M H_2O_2 SOLUTIONS AT 85° C.

Material	Pit Count per sq. cm.	Maximum Pit Depth (mils)	Penetration Rate (mil/mo)	Weight Loss (MDM)
Be Lot 1	1.9	.8	0.059	27.8
356 Al.	2.2	.8	0.003	2.0
Be Lot 1	0.3	1.2	0.046	22.7
356 Al.	1.3	1.2	0.008	5.1
Be Lot 2	5.7	1.7	0.017	7.9
356 Al.	0.1	3.6	0.016	10.9
Be Lot 2	1.8	3.5	0.022	10.3
356 Al.	1.4	25.9	0.011	7.9

The overall corrosion rate of the Lot 1 samples coupled with 356 aluminum again shows an increase of from 2 to 4 fold over that of samples exposed alone. The beryllium was anodically attacked.

With the Lot 2 - 356 aluminum couples, there is no definite electromotive potential difference shown. The rate of attack on both the beryllium and the aluminum is slightly higher in the couple tests than in individual metal tests. The only logical explanation for this development is, that fluctuations in solution could cause reversals of polarity, making the beryllium anodic one time and cathodic another. Thus the high rate of attack during the anodic periods might increase the long term average rate.

The pitting frequency of all specimens was higher in the coupled than in the uncoupled tests. Figure 5 shows the effects of coupling quite clearly.

8.3 Beryllium - 356 Aluminum Couples (Con't.)

8.32 Effect of High Hydrogen Peroxide Concentrations (Con't.)

No. 1 and No. 1-A are the two flat surfaces of a specimen of Lot 1, QM beryllium exposed alone for 60 days to 0.005M H_2O_2 solution.

No. 2 and No. 2-A are similar surfaces from another sample from the same lot exposed to the same corroding medium, but coupled with 356 aluminum. No. 2 is the surface in contact with a sample of the 356 aluminum. No. 2-A is the opposite face where intimate coupling was only in the center area with the nut and bolt of 356 aluminum.

9.0 THE EFFECTS OF POOR DEMINERALIZED WATER

In the interim between the receipt of samples falling in Lots 1, 2, 3 and 4 and the shipping of the two large pieces of QRM beryllium labelled as Lot 5 the corrosion group had moved from X-10 to new facilities at Y-12. This move entailed a change in our source of water supply. Typical analysis of the water from both sources are listed in Table 10.

These analysis are given here in that it is believed that the changes in contaminant concentrations particularly the increases in Fe, Cl, and SO_4 and the decrease in Specific Resistance influenced the corrosion rate of the Lot 5 samples. This reasoning is based on the fact that a number of extruded samples which were originally tested in X-10 water showed a marked increase in rate of attack when transferred to Y-12 demineralized water. Efforts are being made to establish definite water analysis limits.

9.1 Fabrication History and Chemical Analysis of Lot 5 QRM Beryllium

Two billets, Nos. Y-4405BP RM (A) and Y 4405 BP RMB, 3 1/4" x 3 1/4" x 24", were formed by the standard hot pressing technique from Brush process QRM powder. Samples 1" x 1" x 2 1/4" were cut from these billets for corrosion specimens, while the remainder of each billet is to be formed into standard MTR reflector assemblies. Chemical analysis of these bars cut from a single slab is reported as follows:

Elements	In PPM
Ag	a .5
Al	1900
Cd	< .2
Co	< 1
Cu	62

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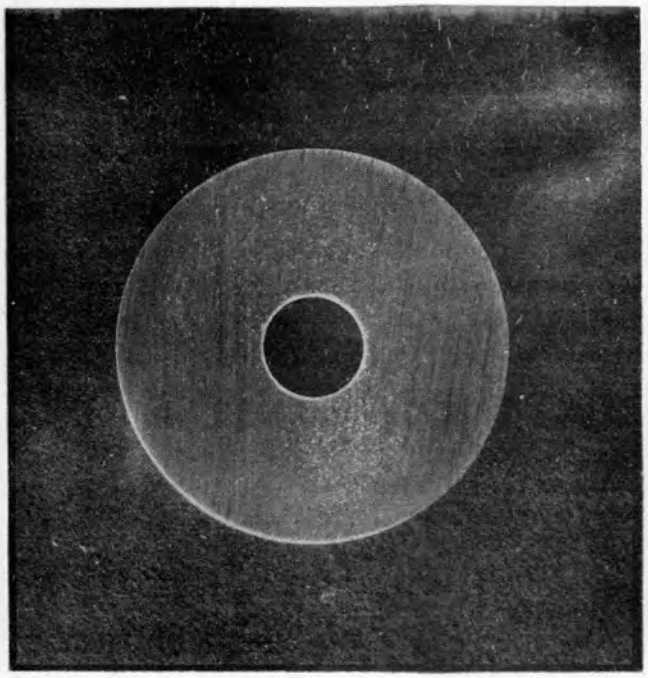
6538



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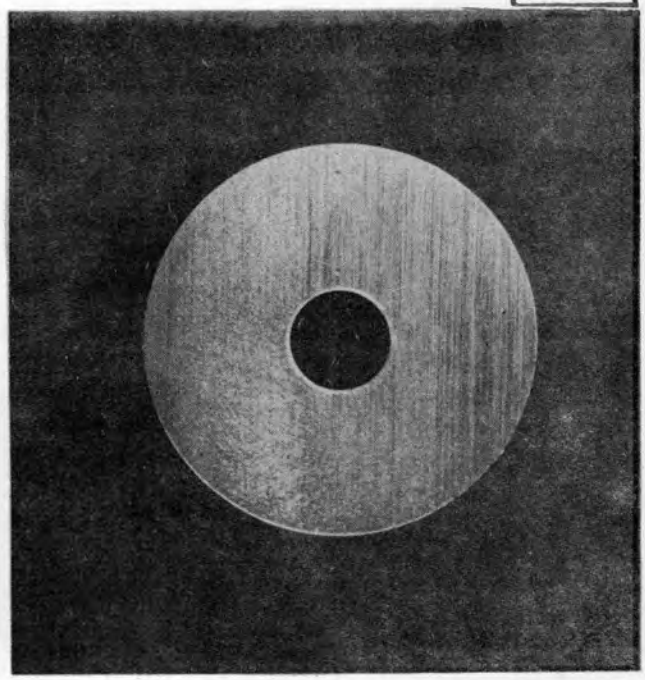

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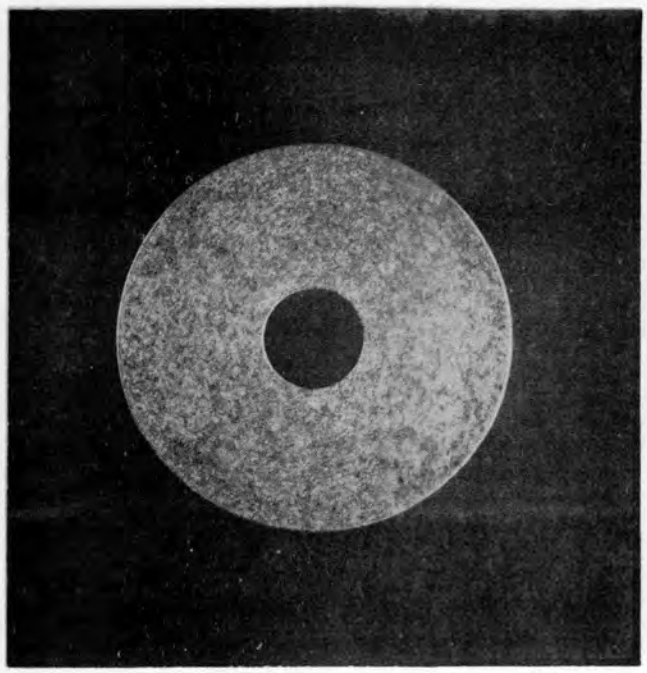

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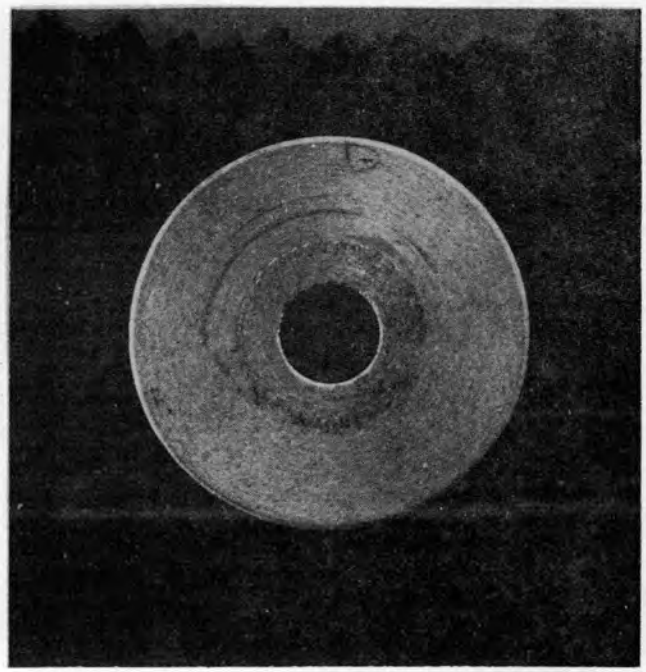

No. 2-A

Magnification 2X

UNCLASSIFIED

FIGURE 5

Effects On Corrosion Of Coupling
QM Beryllium and 356 Aluminum

9.1 Fabrication History and Chemical Analysis of Lot 5 QRM Beryllium (Con't.)

Elements	In PPM
Fe	770
Li	.3
Mg	80
Mn	78
Ni	230
Si	345
Zn	a 60

Above values are from the National Spectrographic Laboratory.

	In Percent
Assay	98.34
Al	.12
Fe	.13
Mg	.02
Be_2C	.34
BeO	1.25
Slag	Not determined

Above values from Brush Beryllium Company.

[redacted]

TABLE 10

TYPICAL ANALYSIS OF DEMINERALIZED WATER FROM TWO SOURCES

Constituents	Analysis X-10	Y-12	Units
pH	5.76	6.05	pH
ppn. Alkalinity as $CaCO_3$	0.0	0.0	ppm
M.O. Alkalinity as $CaCO_3$	3.7	4.0	ppm
Specific Resistance	4.22×10^5	1.59×10^5	ohms
Soap Hardness as $CaCO_3$ data	6.0	11	ppm
Soap Hardness as $CaCO_3$ calc.	1.0	0.0	ppm
Dissolved CO_2	2.0	2.0	ppm
Dissolved Solids	9.1	13.0	ppm
Non Volatile Solids	7.0	6.0	ppm
SiO_2	3.8	1.3	ppm
Fe	0.05	0.20	ppm
Al	0.09	0.03	ppm
Cu	$<$ .02	$<$ .02	ppm
Ni	0.04	$<$ .02	ppm
Cr	$<$ .02	$<$ .02	ppm
Ca	0.4	$<$ .10	ppm
Mg	$<$ .10	$<$ .10	ppm
Na	0.8	2.0	ppm
SO_4	2.0	4.0	ppm
Cl	0.7	4.0	ppm
CO_3	2.2	3.0	ppm
HCO_3	4.5	6.1	ppm

[redacted]

[redacted]

9.2 Corrosion Data on QRM Beryllium in Y-12 Demineralized Water

The following table shows the results of the corrosion tests on Lot 5 QRM Be using the poorer grade of demineralized water.

TABLE 11

STATIC CORROSION OF QRM BE AT 85° C
FOR 60 DAYS DURATION

Condition	Metal	Pit Count Per sq. cm.	Max. Pit Depth (mils)	Penetration Rate (mils/mo.)	Weight Loss (MDM)
1	Be	37	5.6	0.040	18.4
1	Be	47	6.8	0.038	17.9
2	Be	18	4.8	0.069	32.1
2	Be	10	4.8	0.079	36.8
3	Be	29	7.2	0.033	15.4
3	Be	45	6.8	0.047	21.7
4	Be	14	8.8	0.073	33.6
4	Be	23	4.4	0.052	24.0
5	Be	10	6.8	0.066	30.8
	356 Al	.5	2.0	0.05	3.4
5	Be	12	6.0	0.024	11.2
	356 Al	2	5.2	0.060	4.1
6	Be	6	2.8	0.143	66.5
	356 Al	4	6.4	0.010	6.8
6	Be	6	4.4	0.116	53.8
	356 Al	7	7.2	0.019	12.9

Conditions

1) Beryllium as machined in 0.0005M H_2O_2 medium

2) Beryllium as machined in 0.005M H_2O_2 medium

3) Beryllium as machined and annealed 1 hour at 840° C in vacuum exposed to 0.0005M H_2O_2 medium

4) Same as (3) exposed to 0.005M H_2O_2 medium

5) Beryllium as machined coupled with 356 Al (T-7) exposed to 0.005M H_2O_2 medium

6) Same as (5) exposed to 0.005M H_2O_2 medium

Note: All pitting of Beryllium samples tends to spread laterally rather than deepening.

[redacted]

9.2 Corrosion Data on QRM Beryllium in Y-12 Demineralized Water (Con't.)

The pitting frequency in all cases are much higher than the previous tests on QRM metal, although the penetration rates are only slightly higher. However in all cases there were large volumes of corrosion product both on the specimens, and in the solution.

These tests are not considered representative and will be checked in future tests with better water.

10.0 INTERPRETATION OF CORROSION DAMAGE

A complete description of the techniques used in interpreting the corrosion damage may be found in ORNL-298, pp. 109-116. Only a brief summary of these methods is given here.

Evaluation of the damage as concerns the normal corrosion rates, film formation, and galvanic couple behavior is made by three methods:

1) Visual Inspection

i.e. A careful observation of the changes in surface appearance due to corrosion.

2) Measurement of Weight Changes

i.e. A careful weighing of the specimens of known surface area and density, before and after exposure to determine the rates of metal removal and, or, film build up.

3) Microscopic Examination

i.e. The use of the microscope to determine the pit contours and frequency as well as corrosion product appearance.

11.0 DISCUSSION OF RESULTS

The data from these tests indicate three conclusions:

1) That beryllium processed by the two new powder techniques has corrosion resistance comparable with the best vacuum cast and extruded metal tested here.

11.0 DISCUSSION OF RESULTS (Con't.)

2) That more careful control of water purity is necessary to minimize the rate of corrosion of beryllium.

3) Tests in the two conditions on all lots of QM and QRM beryllium show that annealing after machining decreases the corrosion resistance of the metal. This is particularly true of the pitting type of attack.

As all tests with QM and QRM beryllium reported here were carried out under static conditions there is the possibility of further reducing the low maximum penetration rate of .0014 inches per year (extrapolated). This rate is based on the Be 356 Al couple in the poor water.

12.0 FUTURE PROGRAMS

a) Still further testing will be made on the three available types of powder process beryllium, QRM, QMV, and QPT.

b) Sufficient samples from a single lot of vacuum cast and extruded as well as QRM samples are now available to establish maximum allowable ionic constituents; i.e., Fe, Cl, Cu, $(SO_4)^{-2}$.

c) The group will also test the effects of different finishes on the corrosion rate of beryllium. The types to be tested are:

   1) Various ground surfaces

   2) Various milled surfaces

   3) Various milled, finish-ground surfaces

d) The effect of metallic contaminants on the corrosion of beryllium will also be checked. At present they are in test samples with aluminum contents ranging from 0.05% to 1.24% aluminum.

A series of cast beryllium samples from the same base material with the following additions will be made by the Brush Beryllium Corporation:

Impurity	Al	Fe	Si	Be_2C
Amount added	0.2%	0.1%	0.1%	0.10%
	0.4	0.2	0.2	0.25
	0.6	0.4		0.50
	0.8			

12.0 FUTURE PROGRAMS (Con't.)

e) Work is continuing in an effort to establish a basis of comparison between tests at high temperatures and pressures, and long term 85° C tests.

13.0 ACKNOWLEDGMENTS

The writer wishes to express his gratitude to the following group of people for their aid in conducting the tests reported here:

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A. R. Olsen

/tks